IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of Eiju SUZUKI, et al.

Application No.: 10/562,947

Filed: March 13, 2008

For: RUBBER COMPOSITION AND TIRE USING THE SAME

Group Art Unit: 1791

Examiner: Justin R. Fischer

Confirmation No.: 8599

DECLARATION UNDER 37 C.F.R. § 1.132

I, Eiju Suzuki, declare that:

I am one of the inventors of the above-captioned patent application.

I received my Master of Science and Technology from Keio University in 2002, and have been employed by Bridgestone Corporation since 2002, where I have been engaged mainly in research and development of new polymers.

I have made the following experiments in order to evaluate the processability, the wear resistance and the durability of the rubber composition comprising as a rubber component (A) a synthetic polyisoprene rubber having a cis-1,4-bond content of slightly above 99.0% (Polyisoprene rubber D), or having a cis-1,4-bond content of slightly below 99.0% (Polyisoprene rubber E) and (B) a natural rubber, wherein a ratio by mass of (A) the synthetic polyisoprene rubber to a total of (A) the synthetic polyisoprene rubber and (B) the natural rubber is 5-60 mass%.

Experimental Procedure

<Synthesis of Polyisoprene rubber D>

A polyisoprene rubber is synthesized in the same manner as in the Production Example 1 of Polyisoprene disclosed in paragraph [0028] in the specification of the present application except that a polymerization temperature is -10°C. As a result of the synthesis, 39.3 g of a polyisoprene rubber D is obtained in a yield of 92%. In the polyisoprene rubber D, the number average molecular weight (Mn) by a GPC is 975,100 and Mw/Mn is 1.97. As the

micro-structure is determined from an integration ratio of 15.5-16.5 ppm (1,4-trans unit), 18.0-19.0 ppm (3,4-unit) and 23.0-24.0 ppm (1,4-cis unit), the cis-1,4-bond content is 99.1%, the trans-1,4-bond content is 0% and the 3,4-bond content is 0.9%.

<Synthesis of Polyisoprene rubber E>

A polyisoprene rubber is synthesized in the same manner as in the Production Example 1 of Polyisoprene disclosed in paragraph [0028] in the specification of the present application except that a polymerization operation is conducted in a water bath wherein a temperature is about 10°C (lowest temperature: 9°C, highest temperature: 12°C). As a result of the synthesis, 38.2 g of a polyisoprene rubber E is obtained in a yield of 90%. In the polyisoprene rubber E, the number average molecular weight (Mn) by a GPC is 841,200 and Mw/Mn is 2.10. As the micro-structure is determined from an integration ratio of 15.5-16.5 ppm (1,4-trans unit), 18.0-19.0 ppm (3,4-unit) and 23.0-24.0 ppm (1,4-cis unit), the cis-1,4-bond content is 98.9%, the trans-1,4-bond content is 0% and the 3,4-bond content is 1.1%.

(Additional Comparative Examples J, K, L and M)

<Preparation of Rubber composition>

By using the above described polyisoprene rubber D or E, a rubber composition is prepared according to a compounding recipe shown in the following Table D.

<Evaluation of properties of rubber composition>

With respect to the resulting rubber compositions, the processability, wear resistance and durability of the rubber composition are evaluated according to the methods described in paragraphs [0032]-[0034] in the specification of the present application. Results obtained from these experiments are summarized in the following Table D.

Moreover, Table C disclosed in my declaration filed on May 3, 2010 is shown below again for the purpose of reference.

| _ | ٠. |
|---|----|
| Ξ | 긔 |
| 4 | اد |
| | |
| | ٥Į |
| _ | ٦ |
| | |

| | | | Table D | | | | |
|-----------------|---------------------------------|----------|-------------|-------------|-------------|-------------|-------------|
| | | | Additional | Additional | Additional | Additional | Comparative |
| | | | Comparative | Comparative | Comparative | Comparative | Evample 6 |
| | | | Example J | Example K | Example L | Example M | ם אולוושעיד |
| | Natural rubber (RSS#3) | | 95 | 40 | \$6 | 40 | 100 |
| | Polyisoprene rubber D *7 | | 5 | 09 | 1 | • | 1 |
| | Polyisoprene rubber E *8 | | 1 | 1 | 5 | 99 | , |
| | Carbon black *4 | | 20 | 50 | 50 | 50 | 50 |
| Formulation | Stearic acid | parts by | 2. | 2 | .2 | 2 | 2 |
| TOTTE THE TOTAL | Antioxidant 6C *5 | mass | 1 | 1 | - | 1 | |
| | Zinc oxide | | 3 | 3 | دب | 3 | m |
| | Vulcanization accelerator DZ *6 | | 8.0 | 0.8 | 8:0 | 0.8 | 9.0 |
| | Sulfur | | 1 | ·•1. | 1 | 1 | ij |
| | Processability | | 104 | 122 | 104 | 611 | 100 |
| Evaluation | Evaluation Wear resistance | index | 100 | 92 | 100 | 91 | 100 |
| | Durability | | 75 | 80 | 94 | 78 | 100 |
| | | | | | | | ***** |

The polyisoprene rubber synthesized as described above, cis-1,4-bond content=99.1%, 3,4-bond content=0.9%. The polyisoprene rubber synthesized as described above, cis-1,4-bond content=98.9%, 3,4-bond content=1.1%. 00 *

N339, made by Tokai Carbon Co., Ltd. N2SA=93m2/g. *4

N-(1,3-dimethylburyl)-N'-phenyl-p-phenylenediamine, made by Ohuchi Shinkou Kagaku Co., Ltd., Nocrac 6C.

N.N.-dicyclobexyl-2-benzothiazolyl sulfenamide, made by Ohuchi Shinkou Kagaku Co., Ltd. Nocclet DZ.

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| | | | | Table C | C | | | | |
|-------------|---------------------------------|----------|------------|-------------|-------------|------------|-------------|-------------|-------------|
| | | | Additional | Additional | Additional | Additional | Additional | Additional | Comparative |
| | | | Example | Comparative | Comparative | Example | Comparative | Comparative | Example 6 |
| | | | Ą | Example F | Example G | В | Example H | Example I | o aldmayor |
| | Natural rubber (RSS#3) | | \$6 | 95 | 95 | 40 | 40 | 40 | 100 |
| | Polyisoprene rubber A *I | | 5. | • | | 99 | 1 | 1 | , |
| | Polyisoprene rubber B *2 | | • | • | 5: | .1 | t | 99 | ī |
| | Polyisoprene rubber C *3 | | ı | 5 | ı | • | 99 | • | • |
| | | | 50 | 90 | 95 | 50 | 50 | 50 | 50 |
| Formulation | | parts by | 2 | 2 | 2 | 2 | 2 | 2 | 7 |
| | Antioxidant 6C *5 | | - | , — | 1 | 1 | | | - |
| | Zinc oxide | | 8 | £ | 3. | 3 | 'n | 3 | 3 |
| | Vulcanization accelerator DZ *6 | | 8.0 | 8'0 | 0.8 | .8.0 | 9.0 | 8.0 | 8.0 |
| | Suffur | | 1 | 1 | 1 | 1 | | 1 | |
| | Processablity | | 801 | 1.04 | 103 | 140 | 124 | 117 | 100 |
| Evaluation | Evaluation Wear resistance | index | 101 | 100 | 100 | 86 | .94 | 91 | 100 |
| | Durability | | 1.6 | 166 | 93 | 06 | 82 | 77 | 100 |

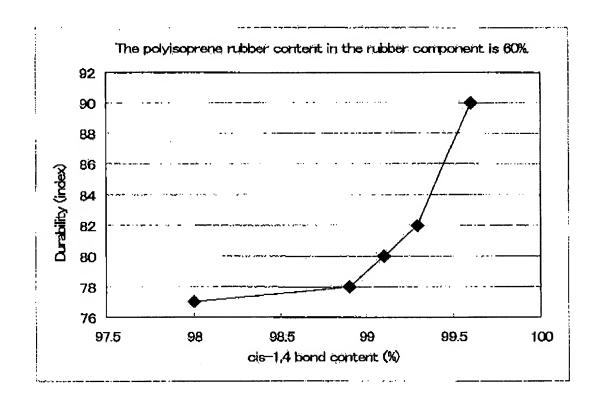
Produced by the Production Example 1 of Polyisoprene described in paragraph [9028] in the specification of the present application, cis-1,4-bond content=99.6%, 3,4-bond content=0.4%. -

IR2200, made by JSR Corporation, cis-1,4-bond content=98.0%, 3,4-bond content=2.0%.

Produced through the method described in my declaration filed on January 13, 2010, cis-1,4-bond content=99.3%, 3,4-bond content=0.7%. N339, made by Tokai Carbon Co., Ltd. N₂SA=93m²/g. * (r)

N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine, made by Ohuchi Shinkou Kagaku Co., Ltd., Nocrac 6C. N,N'-dicyclohexyl-2-benzothiazolyl sulfenamide, made by Ohuchi Shinkou Kagaku Co., Ltd. Noccler DZ. * *

Further, the results of the durability of each rubber composition are shown in the following graph.



(Summary)

As seen from the above results, when the 1,4-bond content of the polyisoprene rubber exceeds 99%, the durability of the rubber composition is improved.

Further, when the 3,4-bond content is not more than 0.5%, the durability of the rubber composition is highly improved as seen from the results of Additional examples A and B in Table C.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code and that such willful false statements may

jeopardize the validity of the application or any patent issuing thereon.

Date: 2-4-20// Declarant: Eiju Suzuki